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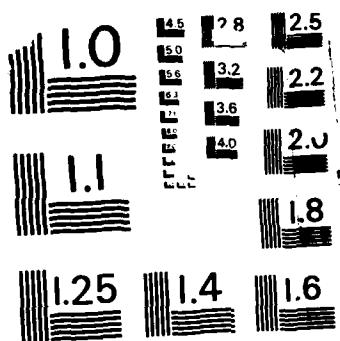
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Analysis of Component Contamination from Accelerated Contamination Testing of Five-Year Vuilleumier Cryocooler

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This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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→ Unit S2 of the Hughes Vuilleumier Cryocooler Program has undergone accelerated contamination testing at twice the service speed and approximately three times the service pressure expected in a normal unit. Debris samples were obtained from all areas of the cooler; samples of regenerator balls were collected from the second and third stages only. The debris has been characterized by an investigative analysis using the analytical techniques of X-ray diffraction, Fourier transform infrared spectroscopy (FTIR), ion		

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microprobe mass analysis (IMMA), and scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDXS) with image analysis. Most of the particulates appear to be metal fluorides except on the first and second stage cold rider areas where silicates predominate.

lead

When observed in the SEM the balls appear to have a film on them. When examined in the IMMA, Si, F, Pb, Sn, and Sb can be identified in the second stage while in the third stage, Si, F, Pb, and Sb are present. In addition, Fe and Cr were observed on both samples. All of the analyses are consistent with a corrosion mechanism of wear.

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I. INTRODUCTION

Recently, accelerated life testing of unit S2 of the Hughes Aircraft Company's Five-Year Vuilleumier (VM) Cryocooler was completed. For the test, the unit was operated for a total of 9710 hours at twice the speed and approximately three times the pressure expected of a unit in actual service. Several stoppages and working fluid exchanges occurred during the course of the test which might have affected the overall performance of the cooler. These stoppages have been described in several Hughes reports.¹⁻³ The S2 unit has been dismantled for overall inspection and analysis of wear debris generated during the test. Aerospace Corporation Materials Sciences Laboratory personnel obtained a group of powder samples (see Table 1) for analysis by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDXS) and image analysis (IA), and ion microprobe mass analysis (IMMA). In addition, samples of the regenerator balls from the second and third stages were obtained for examination solely by IMMA.

II. EXPERIMENTAL PROCEDURES

Two methods were used for obtaining powder debris samples: (a) abundant deposits were scraped off surfaces onto pieces of waxed weighing papers which were then carefully folded and stored in plastic containers, and (b) sparse deposits were removed from surfaces with Scotch® magic mending tape and placed face-up in plastic containers. Optical photographs were taken of each of the samples collected from the internal cryocooler surfaces and the location of each sample was documented (Table 1).

Each of the eight large, granular samples was divided into several fractions for study by different analytical methods. X-ray diffraction analysis was performed first. Samples were finely ground in a mortar, poured into glass capillaries, and placed in a Debye-Scherrer camera. The camera was mounted on a Philips 3100 XRD unit, and copper $K\alpha$ radiation was used to irradiate the samples for several hours. The diameters of the diffraction rings on the resultant films were measured and converted to interplanar spacings. The materials producing the patterns were identified by computer matching with the JCPDS X-ray diffraction data file. This method is based on crystal structure of materials, so that both compounds and elements present in the unknowns were identified.

The Nicolet MX-1 FTIR spectrometer was employed to identify functional groups and bonding arrangements in the powdered samples. The 4X beam condenser attachment was mounted on the FTIR spectrometer and a thin sodium chloride plate was inserted across the 0.5 mm aperture of the sample holder. This was scanned by the IR beam in order to obtain a background spectrum. The unknown particulates were then mounted on the NaCl plate by means of a small Ti probe. The spectrometer chamber was gently purged with N_2 for several minutes to remove CO_2 and water vapor. Samples were scanned for a period of four minutes (128 scans). The information obtained was computer-averaged and the spectra which appeared on the CRT screen were deciphered by comparison with IR plots of standards.

Table 1. Debris Samples Obtained from the S2 Unit

Sample No.	Location
1	Top of drive mechanism
18	2nd stage cold rider area
20	1st stage cold rider area
26	3rd stage cold rider area
40	Hot cylinder #2 near rider
47	Hot cylinder #1, liner bore, 1 in. from hot end
48	Hot cylinder #1, liner bore, midway from hot end
51	Hot cylinder #1, ambient end of hot displacer

IMMA analysis was performed on the deposit taken from the liner bore, midway from the hot end, hot cylinder No. 1, in order to establish the absence or presence of fluorine. A small amount of powder was pressed into an ultrapure pellet of In on a standard 1-in. Al sample mount. This was placed in an IMMA holder and inserted into the instrument chamber. After evacuation of the chamber, a primary beam of $^{18}_2O^+$ ions, accelerated to 20 kV, was focussed on the sample, causing sample material to be sputtered off. A fraction of the sputtered material was ionized and the positive secondary ions mass analyzed by the magnetic spectrometer. The intensity of the characteristic peaks was recorded as a function of mass; they were identified from comparison with tabulated values.

All the powders and tapes were examined using the JSM 840 SEM. The 9900 EDAX analyzer was used to identify elements present in the various samples, and the Lemont image analysis system was programmed to measure particle sizes in excess of 0.3 μm . Powder samples were liquid-dispersed on a filter paper, dried, and mounted to 1-in. carbon sample holders with double-back tape. Tape specimens were cut into small squares and secured to C stubs with carbon cement, Aquadag. All samples were carbon-coated in order to reduce the effects of charging when exposed to the electron beam. SEM photographs were taken of each sample using an accelerating potential of 10 kV. Elements in each sample were identified by the position of their characteristic K or L X-ray energy peaks.

The regenerator balls obtained from the second and third stages were also analyzed in the IMMA but were affixed to an Al sample mount with Ag paint instead of In. Operating conditions for the IMMA were the same as those used for the powder debris.

In the SEM/EDXS/IA, the elements examined were: Na, Mg, Al, Si, P, S, Cl, Cd, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Pb and Br. This general element list was used because of the time constraints involved in creating a new chemistry file program in the IA. It does not include all of the elements expected to be present and includes some which are not expected to be present. The Na, Mg, Al, Si, P, K, and Ca are expected to be present in the glassy particulates detected while Ti, V, Cr, Mn, Fe, and Ni may be present in the metallic components. The S component is indistinguishable from Mo and further analysis of the samples would be required for particles containing this component.

III. RESULTS

The Debye-Scherrer films are not reproduced in this report but the results are as follows: Sample 1 has been identified as Co_5Sm while in samples 20, 26, 40, 47, 48, and 51 the primary constituent is $\text{Fe}_2\text{F}_5 \cdot 7\text{H}_2\text{O}$. This material could not be distinguished unequivocally from the Ni or Cr fluoride materials which may also be present.

The IMMA pattern for sample 48 is shown in Figure 1. The elemental peaks which have been identified are F, Na, Mg, Al, Ca, Ti, Cr, Fe, Ni, and In (from the sample mount). The remaining peaks are associated with various combinations of these elements.

A typical FTIR scan is given in Figure 2 and appears to be that of a fluorinated organic compound. All of the FTIR scans were similar. The SEM/EDXS/IA results for all the samples except sample 1 showed the presence of Fe, Cr, and Ni. In addition, samples 18, 20, and 47 exhibited evidence of Si, Ca, Na, Al, Mg, K, and P while samples 40, 47, 48, and 51 appeared to contain S(Mo). The particle sizes observed varied from 0.25 to 10 μm with the majority of particles measuring between 0.5 and 3 μm .

The IMMA patterns obtained from the regenerator balls are shown in Figures 3 and 4 for the second and third stages, respectively. Both patterns show Si, F, Pb, Sb, Fe, and Cr. The pattern for the second stage also contains Sn.

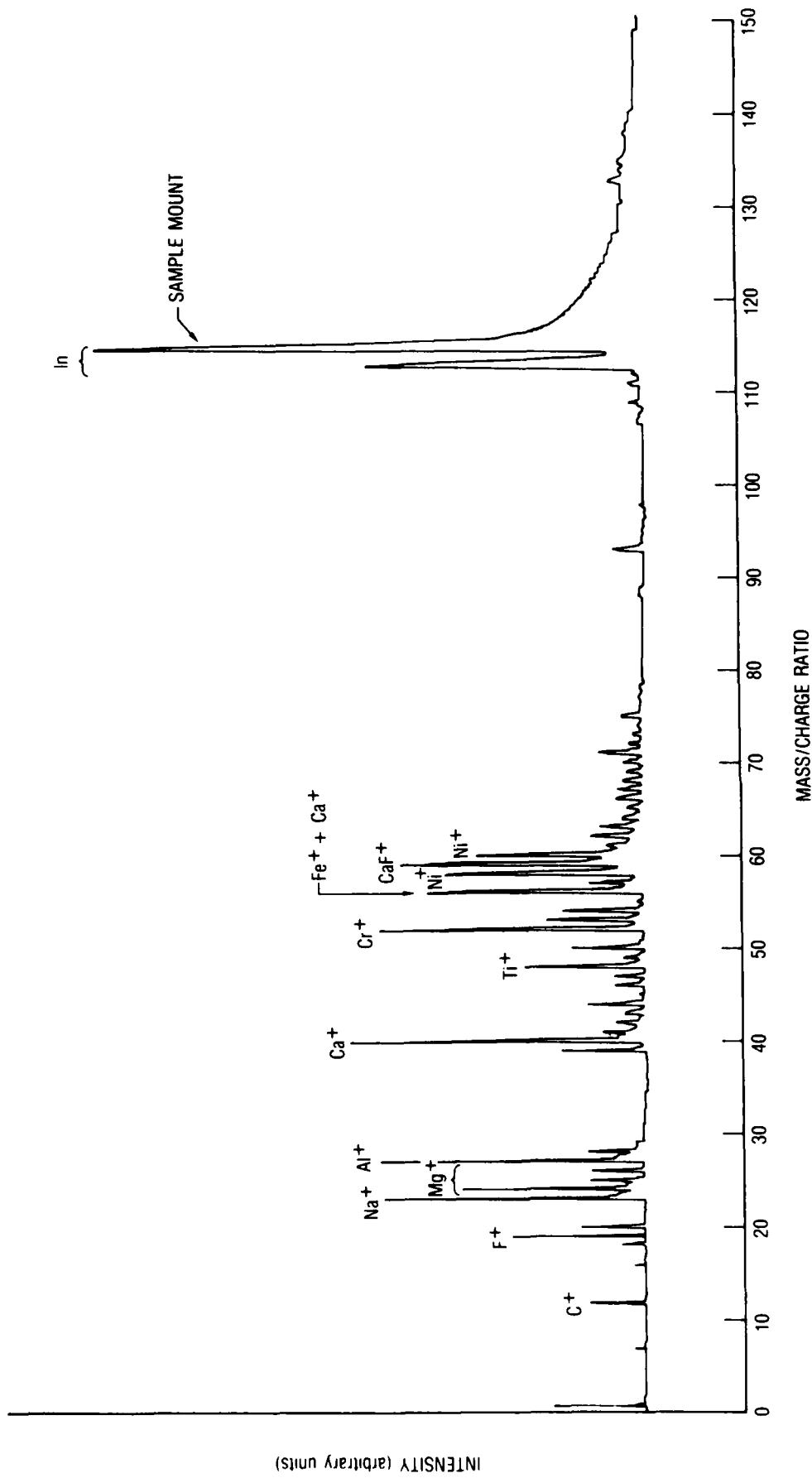


Figure 1. IMMA pattern for sample 48, taken from liner bore of hot cylinder #1, midway from hot end.

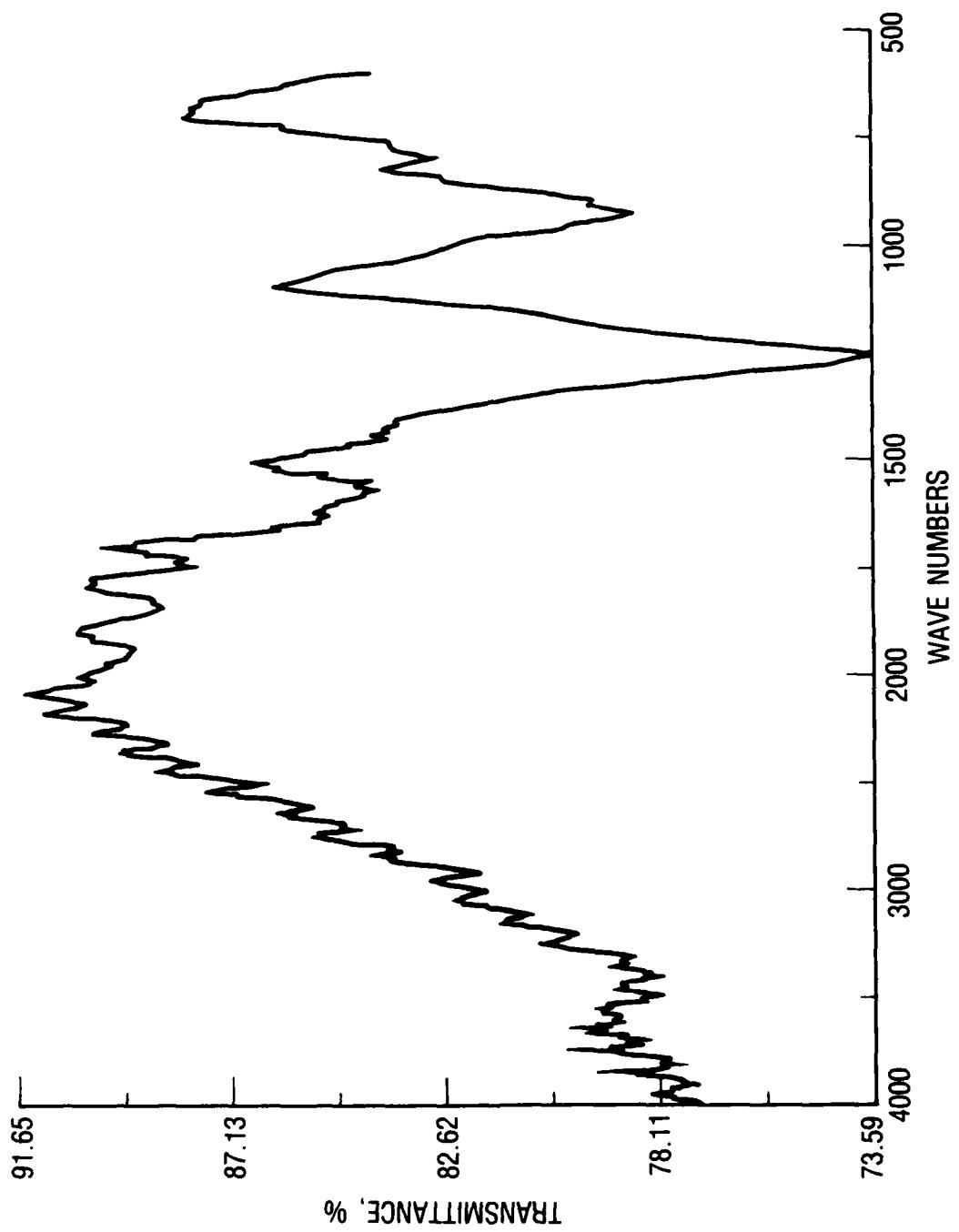


Figure 2. FTIR scan of sample 18, taken from the second stage cold rider area.

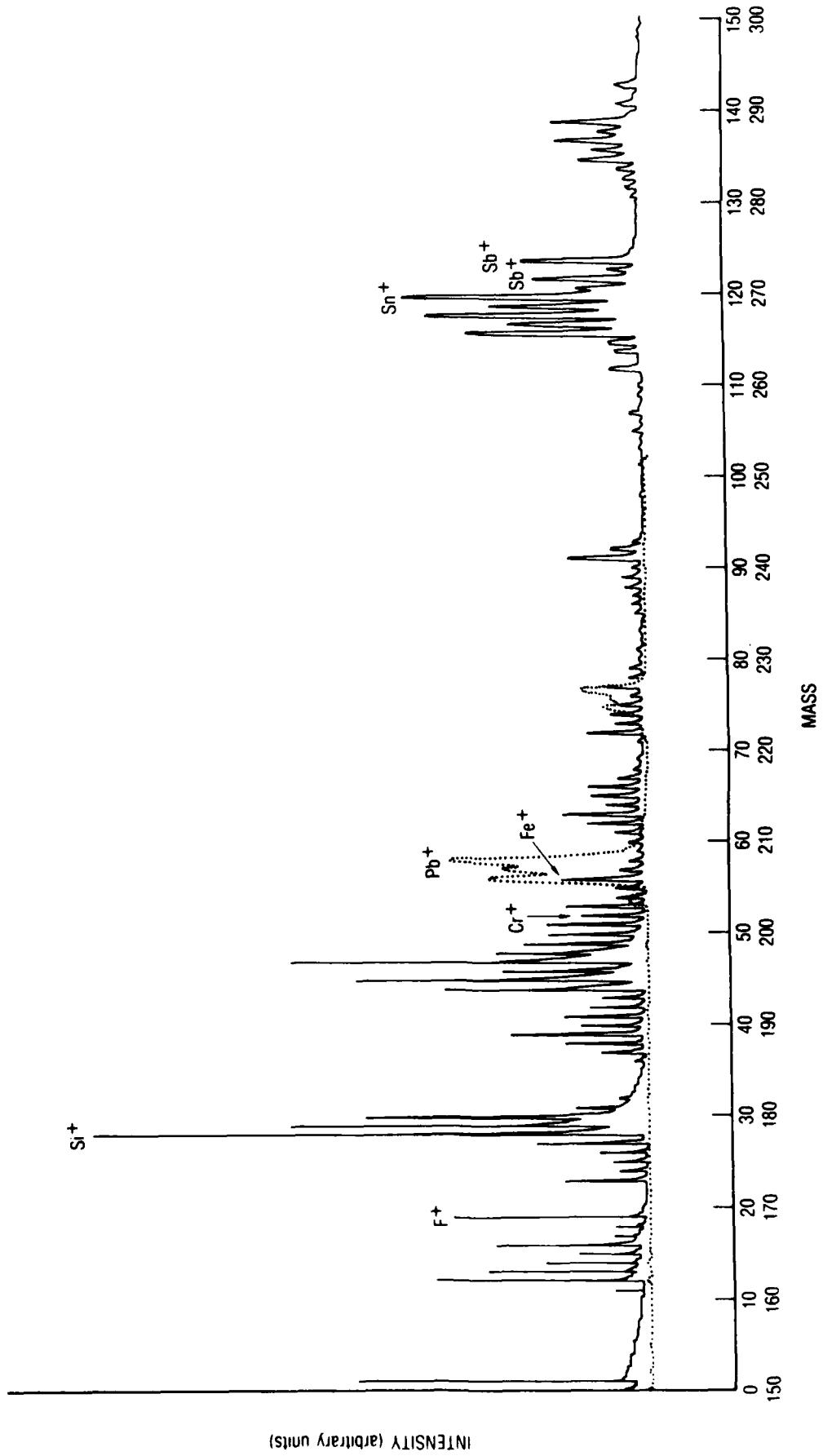


Figure 3. IMMA pattern for second stage regenerator balls. Mass/charge 150 = 300 shown by dotted curve.

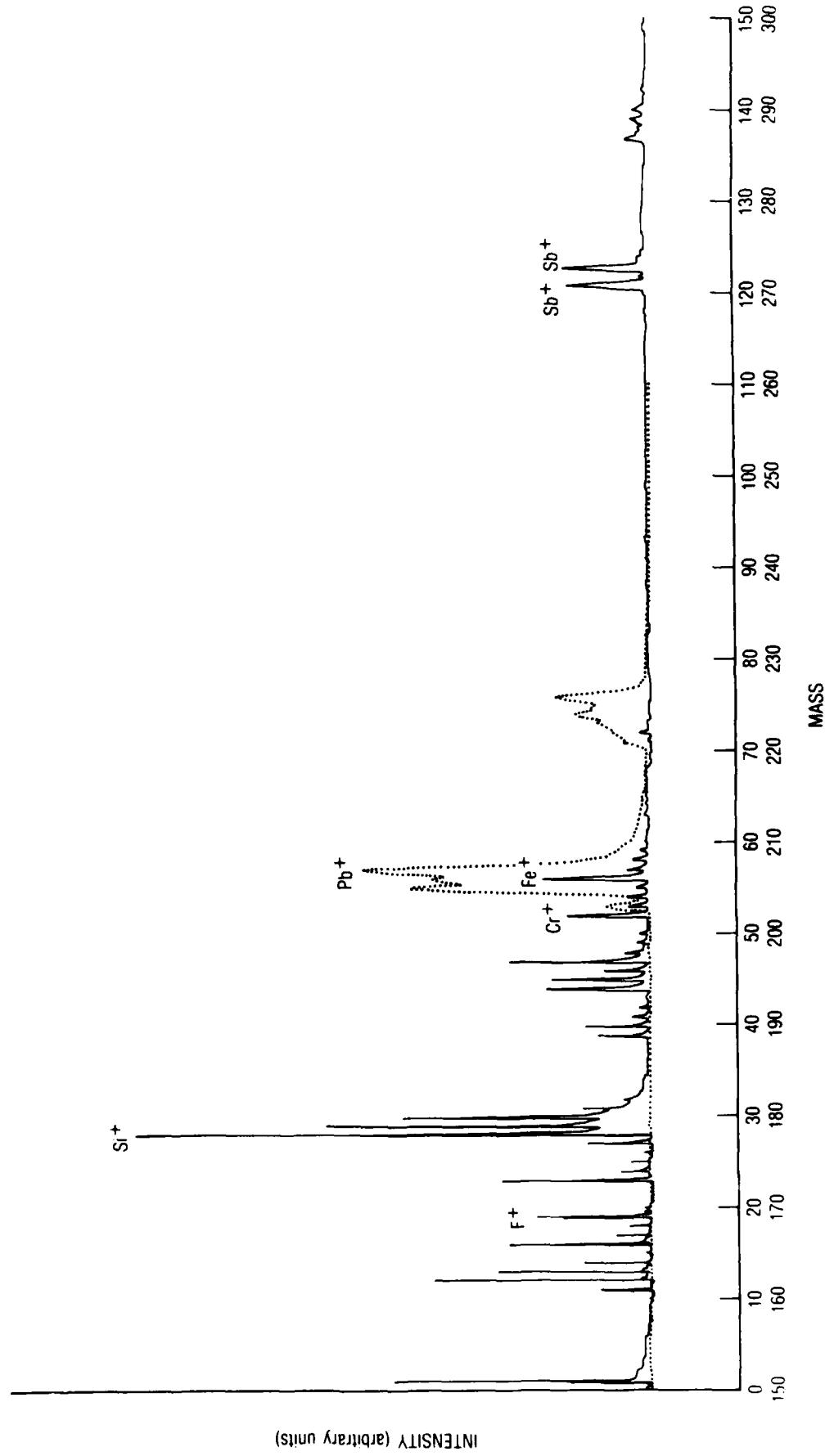


Figure 4. IMMA pattern for third stage regenerator balls. Mass/charge
 $150 = 300$ shown by dotted curve.

IV. DISCUSSION

Sample 1, taken from the top of the drive mechanism, was examined only by XRD and was identified as $Co_5 Sm$. This magnetic material is part of the drive mechanism.

Samples 18, 20, and 26 are from the cold end of the unit (see Table 1). All three samples contain substantial amounts of silicates (determined by SEM/EDXS/IA) with the greatest percentage of silicates appearing in samples from the second stage. The silicates are part of the rider/seal material, Fluorogold, which is a glass-filled Teflon. The SEM/EDXS/IA results also showed substantial amounts of metal-containing compounds which were found to be metal fluorides from XRD studies. The primary metals present were Cr, Ni, and Fe which are the major constituents of Inconel 718 and 321 stainless steel bore materials. The nominal compositions for these materials are given in Table 2.

The metal fluorides probably result from a chemical reaction between the F of the Teflon and the bore materials. At this time, it is not entirely clear how the F was generated. The sole source of F in the system, however, is the Fluorogold and the mechanism must involve degradation of this material, either by abrasion or heat.

Samples 40, 47, 48, and 51 are from the hot cylinders of the unit. Sample 40, from around the rider area, was found to be a metal fluoride. Metallic elements identified were Ni, Fe, Cr, and Mo, consistent with the metal liner bore of Inconel 718. In hot cylinder #1 (sample 47), near the hot end, the same metallic elements appeared, combined as oxides as determined by XRD analysis. There were also some silicates present in this sample. At both the ambient end of the hot displacer and midway up the liner bore, only metallics were identified. The X-ray results were inconclusive.

All the samples contained some organic material which appeared to be the same species in all locations. Delrin AF (a fluoropolymer fiber reinforced acetal homopolymer) is present in the system at the inlet assembly rider, while Fluorogold is present at several locations. The unknown organic material is believed to be a derivative of Fluorogold rather than Delrin.

Table 2. Nominal Compositions of Metal Alloys
Used in Wearing Parts of S2

Inconel 718	321 Stainless Steel
54.6 Ni	72 Fe
19 Cr	18 Cr
17 Fe	10 Ni
5 Nb	+ Ti
3 Mo	
0.8 Ti	
0.6 Al	

Inconel 718 is used to fabricate the bore of the 1st stage cold end and hot cylinder liner bore. 321 stainless is used to construct the 2nd and 3rd stage cold end bores.

The majority of particles were in the 0.5 to 3.0 μm range and did not vary significantly from place to place in the cryocooler.

The second stage regenerator balls were 100 μm in diameter and were coated with substantial amounts of Si and F in addition to Pb, Sn, and a small amount of Sb. The regenerator balls from the third stage were larger (250 μm diameter) than those in the second stage and also showed large amounts of Si and F. These balls also contained Pb and Sb but not Sn. In both IMMA spectra, there was evidence of Fe and Cr also in addition to the other elements detected.

It was noted above that the cold stages contained Fluorogold (glass-filled Teflon) which would be a likely source of both the Si and F deposited on the balls. Fe and Cr are components of the liner bore material (321 stainless steel) while Pb, Sb, and Sn are parts of the alloy composition of which the pristine balls are made. The stability of fluorinated Pb and Si-Pb materials relative to the initial composition has not been determined. The presence of Si and F on the ball surfaces, however, may significantly change the heat transfer characteristics of the regenerator.

V. SUMMARY AND CONCLUSIONS

Debris samples from the Hughes VM cryocooler unit S2 have been analyzed. The debris consists of silicates, metals, and metal fluorides. These materials correspond to the glass in the seals and riders, liner bore and bore materials, and reacted metals from the bore areas. The mechanism of degradation of the Fluorogold (source of F) is not clear at present but should be investigated. Organics are also present which appear to correspond to material from the Fluorogold.

Both Si and F have been detected in significant amounts on the surface of the regenerator balls of the second and third stages of the Hughes VM cryocooler unit S2. The presence of these elements may degrade the performance of the regenerator during operation.

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3. Ibid, Part XI, July 1984.

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